

Los Alamos National Laboratory
Environmental Restoration Program
Standard Operating Procedure

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**Clay Mineral Separation for
X-Ray Diffraction Analysis**

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CLAY MINERAL SEPARATION FOR X-RAY DIFFRACTION ANALYSIS

1.0 PURPOSE

The purpose of this procedure is to describe separation of clay minerals from bulk rock samples and preparation of oriented sample mounts of clay minerals for analysis by X-ray diffraction.

2.0 SCOPE

2.1 Applicability

The procedure applies to separation and X-ray diffraction investigation of clay minerals for the Environmental Restoration (ER) program, and the ER certified user has the responsibility to adhere to this procedure when conducting clay mineral separations for the ER program.

2.2 Training

Personnel using this procedure for the ER program must have an understanding of the objectives of this procedure and be trained to use it and the SOPs in Section 1.0, General Instructions. Reading and understanding the procedure shall constitute training to this procedure.

3.0 DEFINITIONS

N/A

4.0 BACKGROUND AND/OR CAUTIONS

This procedure is based on Stokes Law settling of particles in a solution.

The Ultrasonic probe is placed in a sound-dampening box to help protect the hearing of the operator.

The use of de-ionized or distilled water to wash and disaggregate the samples is necessary to ensure that no chemical alteration of the clays occurs due to cation exchange. Thoroughly clean all sample preparation equipment and work areas before use.

Adherence to this procedure results in an acceptable sample for X-ray diffraction analysis. The purity of the individual samples and their size fractions are determined by X-ray diffraction. It is up to the certified person conducting the separation to determine if the clay fractions are of adequate purity for their work and shall be documented on the Daily Activity Log (see SOP-01.04). There are no real potential sources of error since this is a qualitative procedure.

5.0 EQUIPMENT

- Shatterbox (or large ball mill) -- most all acceptable.
- Ultrasonic probe capable of ~200 W.
- Centrifuge capable of 8000 RPM with a Sorvall GSA head.
- Sorvall GSA centrifuge head.
- Sorvall SS-34 centrifuge head (optional).
- Spray Drier (optional).
- No software is used.

6.0 PROCEDURE

6.1 Calibration

Not applicable. Samples are examined for purity using X-ray powder diffraction in accordance with SOP-09.03.

6.2 Samples

Samples will be tracked, handled, shipped, and stored in accordance with the procedure for Sample Identification and Control for Mineralogy-Petrology Studies (Ref A). Great care shall be exercised not to mix or contaminate samples. All subsequent analyses rely on this procedure. Carefully label containers into which the sample separates will be placed. Label side and top of each container so that tops cannot be switched.

6.3 Procedural Deviations

Deviations from this procedure shall be fully documented on the Daily Log by the person conducting the work and shall explain the deviation and the effects it may have on the resulting work.

6.4 Separation Procedure

6.4.1 Obtain approximately a one-to-two-inch slice of drill-core or an equivalent amount of drill cuttings or bulk-rock. If necessary, smaller amounts of sample can also be processed using this procedure.

6.4.2 Rid the sample of surface contamination using one of the following:

- Brisk strokes of a clean, dry bristled brush.
- Rinse with de-ionized or distilled water and dry thoroughly -- preferably at room temperature.

6.4.3 Break the bulk sample down into smaller pieces (less than 0.25 inch in diameter using:

- A mortar and pestle or,
- A mortar and pestle/hydraulic press apparatus.

6.4.4 Powder sample using either a shatterbox or large ball-mill.

6.4.5 Combine the entire powdered sample and mix thoroughly to ensure homogeneity. Place in a bottle properly labeled with the sample identification.

6.4.6 Take approximately 20-30 grams of the powdered sample and suspend it in approximately 500 to 700 ml of deionized or distilled water contained in a large plastic (~1000 ml) beaker. Disaggregate the sample for approximately 10-20 minutes at ~200 W using an ultrasonic probe. Longer times may be used if samples are difficult to disaggregate. Small amounts of sample may be readily separated using this procedure by substituting appropriately scaled glassware.

6.4.7 Remove the beaker containing the suspended sample from the ultrasonic probe enclosure and cover the beaker. Place the beaker on a vibrationally stable surface and allow the sample to settle gravitationally without interruption ideally for a time equal to 47 minutes per cm depth of water. This will allow sedimentation of all particles greater than 2 μ m diameter. For a typical 14 cm water depth, total settling time should be about 10.9 hours. Overnight settling using a water depth of 14 cm will result in sedimentation of slightly smaller particles, e.g., 15 hours settling will sediment particles >1.75 μ m and 20 hours settling will sediment particles >1.5 μ m.

6.4.8 Decant or syphon the supernatant from the beaker into large centrifuge tubes, taking care not to disturb the sediment at the bottom of the beaker. The sediment in the beaker may be discarded.

6.4.9 Place the centrifuge tubes in the centrifuge and separate the ~2.0-0.35 μ m size fraction from suspension at a centrifugation speed of ~5000 revolutions per minute ~5 minutes.

Superspeed Angle Rotors: GSA and SS-34; Ref B).

6.4.10 Remove the centrifuge tubes from the centrifuge and decant the supernatant into large receiving centrifuge tubes, taking care not to disturb the sediment ($>0.35\ \mu\text{m}$ in diameter) at the base of the tube. Save this sediment fraction if needed.

6.4.11 Place the centrifuge tubes containing the cloudy supernatant in the centrifuge and separate the fine clay fraction ($\sim 0.35\text{--}0.10\ \mu\text{m}$ in diameter) at a centrifugation speed of ~ 8000 revolutions per minute for a spin time of 40 to 60 minutes.

6.4.12 Remove the centrifuge tubes from the centrifuge and decant the supernatant into labeled beakers.

6.4.13 Remove the fine clay sediment fraction ($\sim 0.35\text{--}0.10\ \mu\text{m}$ in diameter) from the base of the centrifuge tubes using a spatula and, if necessary, a small amount of deionized or distilled water. Save in a properly labeled sample bottle.

6.4.14 There are several options for processing the remaining supernatant material from step 6.4.13, (this supernatant contains the $<0.10\ \mu\text{m}$ fraction). The options are as follows:

- 1: If not needed, it may be discarded.
- 2: Place the beakers on a low-temperature hotplate or warm surface to evaporate/concentrate until it will fit into a labeled sample bottle.
- 3: It can be further centrifuged using the Sorvall SS-34 head at a speed of ~ 15000 RPM for ~ 1 hour to remove some of the remaining clays.
- 4: It can be run through a spray drier which evaporates the liquid and allows the clay to be collected.

6.5 Preparation of Oriented Mounts for XRD Analysis

6.5.1 Suspend a clay mineral fraction in a small amount (approximately 10 to 20 ml) of deionized water contained in a small beaker.

6.5.2 Thoroughly clean and dry a glass or quartz slide appropriately sized for the diffractometer sample holder being used, and place it on a level, stable, warm surface. The sample number is written with indelible ink on the back of the slide.

6.5.3 Drop the suspended clay sample, using an eye dropper or pipette, onto the slide using only enough sample to cover the slide. Do not overfill the slide and do not add additional suspension after the suspension on the slide has begun to dry to ensure the best orientation of clay particles possible.

6.5.4 Allow the sample to dry undisturbed on the slide.

6.5.5 The sample is now ready for X-ray diffraction analysis (Ref F).

6.6 Clay fractions shall be placed in appropriately labeled sample bottles.

7.0 REFERENCES

Los Alamos National Laboratory Yucca Mountain Project Quality Assurance Manual in Effect January 1990, TWS-EES-DP-101: Sample Identification and Control for Mineralogy-Petrology Studies.

Sorvall Superspeed Angle Rotors, GSA and SS-34 Instruction Manual DuPont Company, Biomedical Products Division, Newtown, CT 06470.

LANL-ER-SOPs in Section 1.0, General Instructions.

LANL-ER-SOP-09.03, Operation of Siemens X-ray Diffractometer.

8.0 RECORDS

8.1 Full records of a clay separation, including full sample name or number, date of separation, and signature of the person separating each sample are to be recorded on Daily Logs.

8.2 All documentation requirements imposed by other procedures, both Yucca Mountain and ER, used during this procedure shall be completed in accordance with the procedures and attached to the Daily Logs.

9.0 ATTACHMENTS

N/A.